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Effect of particle size on the formation of Ti_2AlC using combustion synthesis

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Abstract

This paper provides an insight into the effect of particle size of elemental metal powders and carbon source on the formation mechanism of Ti_2AlC MAX-phase ceramic produced by self-propagating high-temperature synthesis (SHS). The effect of titanium, aluminium and carbon particle size on the $2Ti + Al + C \rightarrow Ti_2AlC$ reaction, the phase evolution of the final product and the porosity in both the green body and product has been examined. The effect of the carbon source in the form of graphite, carbon black and short carbon fibres on the reaction mechanism is explained. It is found that the particle size of the titanium and aluminium reactants had little effect on the phases formed but affected the green density of the reactants and the porosity in the final product. The carbon source used in the combustion reaction had an influence on the phases formed by the SHS reaction and was influenced by the dispersion of carbon particles and the titanium-aluminium particle contact.

Keywords: SHS, Particle size, Porosity, Ti_2AlC, MAX-phase, XRD

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1. Introduction

MAX-phase ceramics are a group of ductile ceramic materials with the molecular formula $M_{n+1}AX_n$ (n = 1, 2, 3….), where M is an early transition element, A is an element from the 'A' group of the periodic table and X is either nitrogen or carbon [1]. A particular advantage of these materials is that they are able to maintain their strength at high temperatures. In addition these ceramic materials possess both ceramic and metallic properties; for example some of their important characteristics are low density, high stiffness, machinability, excellent thermal and electrical conductivity and the ability to exhibit a degree of plasticity at elevated temperature [2]. This fascinating combination of properties has led to researchers to consider the technological importance of these materials as a structural ceramic for high temperature applications.

One of the techniques to produce MAX-phase ceramics is by a solid-state combustion reaction also known as *self-propagating high temperature synthesis* (SHS) [3-6]. SHS is a technique developed for the production of engineering ceramics and other advanced materials [7]. The process involves the ignition of a compact powder reactant mixture in air or an inert atmosphere and an exothermic reaction produces sufficient heat that the reaction becomes self-sustaining; this results in the formation of combustion wave that propagates along the reactants to form the final desired product [8]. A variety of advanced materials, such as Ti_2AlC, Ti_3AlC_2, Ni_3Al, Ni_2Al_3, TiAl, Zr_5Si_3 [9], have been synthesised using this form of solid-state combustion synthesis reaction. The advantages of the SHS process are [7, 8] (i) the non-equilibrium behaviour of this process provides the opportunity to produce metastable phases due to the rapid rate of heating and relatively rapid cooling, (ii) the process is energy efficient compared to the traditional solid-state sintering process as the process takes place within few seconds depending on the exothermic nature of the reaction and the only necessary energy requirement is that to initiate the SHS reaction, (iii) the rapid exothermic reaction minimises the production time and (iv) due to the high temperature of the SHS reaction it is possible to produce products of high purity, since as any low boiling point impurities volatilise. However, the gases entrapped in the sample during the SHS reaction and the volume reduction during the reaction leads to the formation of a porous product [7]. However, this can be overcome by the application of external pressure to densify the product shortly after the SHS reaction has taken place [8].

SHS reactions are generally performed using a cold-pressed reactant powder mixture. As a result, the nature of the particle packing of the reactant mixture plays an important role in determining the green density, the thermal conductivity of the reactant mixture, the particle-particle contact and ultimately the microstructure, porosity and phases formed in the final product [10-16].

1.1 Formation mechanism of Ti_2AlC

There are a variety of studies of the reaction mechanism to explain the evolution of the phases that are formed during combustion of Ti-Al-C based materials and SHS processing of Ti_2AlC [17-19]. A number of reaction mechanisms focus on the temperature range associated with the formation of Ti_2AlC and any intermediate compounds formed during the SHS process [18]. Other work [20] has examined the reaction pathway to form the final Ti_2AlC product, which include Reactions 1-3 [18] and Reaction 4 [17];

$Al(s) \rightarrow Al(l)$ (melting of aluminium at 660^0C) (*Reaction 1*)

$Ti(s) + Al(l) \rightarrow TiAl\ (l)$ (below 900^0C) (*Reaction 2*)

$Ti(s) + C(s) \rightarrow TiC\ (s)$ (above 900^0C) (*Reaction 3*)

$TiAl(l) + TiC(s) \rightarrow Ti_2AlC(s)$ (at approximately1200^0C) (*Reaction 4*)

Zhenbin et al. [19] refers to the mechanism as a solution-precipitation process as the TiC that is formed by Reaction 3 dissolves into the TiAl matrix formed by Reaction 2; this results in the precipitation of Ti_2AlC by Reaction 4. Pietzka et al. [17] described the thermodynamics of the reaction mechanism and reported that the peritectic point of Ti_2AlC is 1625±10°C and above this temperature Ti_2AlC decomposes into TiC and Al. The Gibbs free energy, ΔG of Ti_2AlC was found to decrease in magnitude with increasing temperature between 1000^oC and 1300^oC; where ΔG^{Ti2AlC} (1000^oC) = -65.9kJ/mol and ΔG^{Ti2AlC} (1300^oC) = -54.8 kJ/mol. Therefore the optimum reaction temperature for forming the Ti_2AlC phase may be at lower temperatures, e.g. 1000^0C-1200^0C. Hashimoto et al. and Liu et al. [21, 22] described mechanisms to explain the layered structure of these ternary carbides and how the reactant particle packing density influences the mechanism of formation of Ti_2AlC by SHS. The thermodynamics and the adiabatic combustion temperature of the formation of Ti_2AlC by SHS were considered by Bowen et al. in the form of enthalpy versus temperature curves [23].

1.2 Aims of paper

There are limited experimental studies explaining how the particle size of the metal reactants and variation of the particle size and nature of the carbon source plays an important role in reaction mechanism and this is the focus of this work. In this paper we report results from experiments conducted to understand the effect of particle size and carbon source during the formation of Ti_2AlC by a SHS reaction between the Ti, Al and C elemental reactants, as shown in Reaction 5.

$$2Ti + Al + C \rightarrow Ti_2AlC \qquad \textit{(Reaction 5)}$$

The MAX-phase product, Ti_2AlC, belongs to Ti-Al-C [2] system and we will study how one can tune the porosity of the Ti_2AlC formed via SHS by changing the particle size of the elemental reactants and carbon source.

2. Experimental procedure

Elemental powders of titanium of different particle size included Ti-45μm (Goodfellow 99.5% purity) and Ti-25μm (Pi KEM Ltd, 99.9% purity). The aluminium particles size reactant were Al-15μm, Al-20 μm and Al-60μm (Goodfellow 99.9% purity). The carbon sources used were short carbon fibres (mean diameter 3.55μm, mean length 104.14μm), graphite ≤20μm (Aldrich) and lamp black ≤30μm (Inoxia Ltd).

To undertake the SHS reaction, each powder was mixed according to the stoichiometric molar ratio 2Ti:1Al:1C for 10 – 15 minutes using a mortar and pestle. The combination of above mentioned elements of various particle sizes were mixed to obtain the necessary reactant mixture for Reaction 5. A mass of 0.8g of the elemental powder mixture was pressed at room temperature into a compact cylindrical pellet of 13mm diameter in a uniaxial press using a pressure of 1.77kN/mm^2. The thickness of the pellet varied with the different reactant particle sizes used, as seen in Table 1, and is related to degree of packing of the reactant. Field Emission Scanning Electron Microscopy (FESEM) images of the shape and size of the titanium, aluminium and carbon reactant particles are shown in figure 1.

Table 1. Combination of particle sizes used and their green density. Sample diameter was 13mm and mass 0.8g in all cases. Note Ti_3Al, TiC also formed when using Ti-25 µm, Carbon fibre and Al of 15µm and 60µm.

Element, particle size (µm) and carbon source	Mean thickness (mm)	Green density (g/cm^3)	Compounds formed from SHS
Ti-45, Al-15, Carbon fibre (mean diameter 3.55µm and length 105µm)	2.07	29.07	Ti_2AlC, TiC
Ti-45, Al-25, Carbon fibre (mean diameter 3.55µm and length 105µm)	2.12	28.47	Ti_2AlC, TiC
Ti-45, Al-60, Carbon fibre (mean diameter 3.55µm and length 105µm)	1.99	30.29	Ti_2AlC, TiC
Ti-25, Al-15, Graphite ≤20µm	1.79	33.61	Ti_2AlC, TiC
Ti-25, Al-25, Graphite ≤20µm	1.74	34.71	Ti_2AlC, TiC
Ti-25, Al-60, Graphite ≤20µm	1.78	33.92	Ti_2AlC, TiC
Ti-45, Al-15, Graphite ≤20µm	1.68	35.80	Ti_2AlC, TiC
Ti-45, Al-25, Graphite ≤20µm	1.71	35.18	Ti_2AlC, TiC
Ti-45, Al-60, Graphite ≤20µm	1.67	36.02	Ti_2AlC, TiC
Ti-25, Al-15, Lamp black ≤30µm	1.99	30.24	Ti_2AlC, TiC
Ti-25, Al-25, Lamp black ≤30µm	2.01	29.99	Ti_2AlC, TiC
Ti-25, Al-60, Lamp black ≤30µm	1.97	30.59	Ti_2AlC, TiC
Ti-25, Al-25(0.3 mol excess), Carbon fibre (mean diameter 3.55µm and length 105µm)	2.08	29.02	???
Ti-25, Al-25, Carbon fibre (mean diameter 3.55µm and length 105µm)	2.14	28.12	Ti_3Al, TiC

The cold pressed cylindrical pellets were loaded into a custom built combustion rig and ignited using a resistively heated 'U' shaped tungsten wire of 0.5mm diameter using 30A of electric current in an argon atmosphere at a pressure of 2 bar. The phases formed were analysed using XRD (using a Cu target) and the density and porosity of the SHS product was measured according to the BS EN623-2: 1993 standards.

3. Results and Discussion

Table 1 shows how the green density of the cold-pressed reactant pellets vary with the change in the particle size of the elemental powders indicating the porosity of the green body before the reaction process. The green density was measured since it provides an insight into the packing density of the 2Ti/Al/C reactants, since each pellet was mixed and cold-pressed under same conditions. The pellets were subjected to the SHS mode of combustion synthesis and the compounds detected from XRD for the respective combination of particle sizes are also provided in Table 1. The XRD analysis can be seen in figure 2 (graphite source), figure 3 (lamp black source) and figure 4 (carbon fibre source).

It can be observed that, irrespective of the particle size of both the titanium and aluminium, when graphite and lamp black was used as the carbon source, the final products were Ti_2AlC and TiC; see Table 1 and the XRD results in figures 2 and 3. However, when carbon fibre was used the nature of the products formed is more complex (figure 4). As the particle size of the titanium decreases from 45µm to 25µm, Ti_2AlC does not form, rather Ti_3Al is formed which is an intermediate compound formed during the formation of Ti_2AlC; as explained by Ming et al. [18].

The cold-pressed pellets made with carbon fibre have the lowest green density compared to the reactant pellets formed using graphite and lamp black with similar particle size of titanium and aluminium; as seen in figure 5. The cold-pressed pellets made with carbon fibre (black colour bars in figure 5) have the lowest green density compared to the reactant pellets formed using both graphite and lamp black with Ti and Al of the same particle size. This is due to the relatively large size of the carbon fibre source compared to the titanium and aluminium, resulting is poor particle-particle contact that leads to an incomplete reaction between Ti_3Al and TiC, as seen in Table 1. In this case, only Ti_3Al is formed using carbon fibre source and the SHS reaction in not sustained for the formation of other intermediate compounds such as TiAl and TiC, which in turn reacts to form Ti_2AlC.

Figure 6 shows FESEM images showing how the reactant particles are packed when pressed into a green body. It can be seen that since the titanium, aluminium and carbon reactant particles are of different shape and size when mixed and pressed into pellets they form a variety of particle-particle interconnections depending on the relative particle size. For example, there is a point contact in the case of spherical particles (Figure 6e), line contact in the case of elongated particles (figure 6g) and multiple point contact when there is a large contrast between particle sizes (figure 6i). The nature of the powder packing also influence the appearance of the reactant pressed pellet, where the reactant mixtures with small carbon particles (graphite or lamp black) appear black to the naked eye since it coats the larger aluminium and titanium particles. Green body pellets formed with larger carbon fibres appear metallic in colour since the aluminium and titanium particles coat the larger carbon source. The nature of the packing also influences the green density of the cold pressed reactants, as seen in figure 5, which in turn can affect the SHS reaction process, the reactant thermal conductivity and therefore affect the propagation of the SHS combustion wave.

A possible reaction mechanism for pressed reactants made with titanium (25μm) and aluminium (15μm or 25μm) and short carbon fibre can be explained with the schematic in figure 7(a). When short carbon fibres are used as a carbon source, there is a greater degree of titanium to aluminium particle contact due to the shape and size of the carbon (figure 1(g) and figure 7(a)). As a result the green body pellet has a 'metallic' visual appearance due to poor dispersion of the short carbon fibres; this is shown by the particle arrangement representation in figure 7(a). When the cold pressed reactant is heated, due to high metal to metal contact, the thermal conductivity is high and as the combustion initiates and the propagating combustion wave moves across the reactants rapidly. This rapid propagation of combustion wave and poor dispersion of carbon particles, does not favour the completion of the final reaction step for the formation of Ti_2AlC via Reaction 4 but favours a reaction between titanium and aluminium:

$$3Ti + Al \rightarrow Ti_3Al \qquad (\textit{Reaction 6})$$

and TiC by Reaction 3. The adiabatic combustion temperature of Reaction 6 is in excess of 1300^{o}C [24]; and once complete there is relatively rapid cooling of the product after passage of the combustion wave since there is no reaction between TiAl and TiC to form Ti_2AlC via Reaction 4. As a result the XRD of the product formed by this composition indicates Ti_3Al and TiC (figure 4(ii)) as the main reaction product and the reactants pressed with 25μm titanium, 15μm or 25μm aluminium and short carbon fibre (figure 4(ii)) do not form Ti_2AlC.

However, when smaller graphite or lamp black particles are used as the carbon source, the carbon particles are in intimate contact with the titanium and aluminium particles due to the particle size and geometry; see figures 1(f) and 1(h). The particle arrangement in shown in the schematic in figure 7(b). As a result, the cold pressed reactants are both black in colour (this is in contrast to the green body reactant samples obtained using short carbon fibres, which appear metallic). In this case, there is a relatively poor titanium metal to aluminium metal contact as the carbon particles are located between the metal-metal reactant particles as seen in figure 7(b) and also the FESEM images in figures 6(a), (b), (c), (d), (e) and (j). This may also decrease the reactant thermal conductivity and slows down the propagating combustion wave as in figure 7(b). This leads to a slower propagation of combustion wave and ensures completion of Reactions 2, 3 and 4; thereby leading to the formation of Ti_2AlC by the solution precipitation mechanism [19] (Reaction 4). It is observed that in this case there is a slower 'cooling down' stage during the SHS reaction and the sample continues to 'glow' red after passage of the combustion wave due to progression of Reaction 4 to form Ti_2AlC after the combustion wave has passed, and this is more likely to occur in the case of reactant made with graphite and lamp black as seen in the schematic in figure 7(b).

To examine this hypothesis that a combustion wave associated with good particle-particle contact, a less exothermic reaction and slow cooling of the product after the combustion wave has passed favours the formation of Ti_2AlC, the aluminium content in the reaction mixture was increased in the reactant mixture made with short carbon fibres with 25μm titanium and 25μm aluminium. When excess Al (y in reaction 3) is added to the Reaction 1, it becomes:

$$2Ti + (1 + y)\,Al + C \rightarrow Ti_2AlC + yAl \qquad \text{(Reaction: 7)}$$

The effect of diluents such as aluminium and titanium on the combustion reaction of Ti_2AlC was examined by Bowen et al. [23] and it was found that it decreases the exothermicity of the reaction, since the thermal mass of the system is increased. The addition of excess aluminium also served to increase the green density due to the presence of the ductile aluminium phase (see Table 1 and figure 5). Figure 8 is the XRD showing the evolution of compounds when a range of excess aluminium additions is used (y equivalent to 0.1, 0.2 and 0.3). When y=0.3 the final product formed was Ti_2AlC (figure 8) and supports the theory that a higher green body density, higher carbon-metal particle contact and a lower combustion temperature leads to slow cooling of the sample upon the completion of the reaction and favours the formation of Ti_2AlC ceramic by SHS reaction.

Figure 9 shows the porosity of Ti_2AlC product formed with different combination of titanium, aluminium and carbon particle sizes; the porosity values are a mean of seven samples per reactant mixture. It can be observed that Ti_2AlC prepared with graphite is less porous than the other samples and is likely to be due to the higher green body density (see Figure 5) and the complete reaction to form the Ti_2AlC. The products formed using carbon fibres are more porous, again due to the lower initial density of the reactant (Figure 5) and incomplete reaction. This data is useful as it is a correlation to the exothermicity of the reaction and also aids in choosing the reactant particle size and carbon source for forming Ti_2AlC MAX-phase ceramics materials by SHS.

5. Conclusion

This paper has examined the influence of the particle size of the titanium, aluminium and carbon reactant on the products formed and thereby control phase evolution in Ti_2AlC MAX-phase ceramics manufactured by SHS. The particle size of the titanium and aluminium reactant has little effect on the final products formed. However, the shape and size of the carbon reactant particle has an influence on the packing density of the reactants and it was found that the more porous green body sample it is less likely to form Ti_2AlC and forms Ti_3Al and TiC, which is an intermediate compound, formed during the formation of Ti_2AlC. It was observed that high titanium-metal to aluminium-metal contact and large carbon particle size in the reactant pellet favours the formation of Ti_3Al and hinders the formation of Ti_2AlC by precipitation process. It was demonstrated that by lowering the combustion wave velocity by addition of diluents such as excess aluminium, it is possible to control the combustion wave and facilitate the formation of Ti_2AlC. It was also demonstrated that fine-tuning the carbon particle size could control the exothermicity of the reaction and control the porosity of the final product without the application of external pressure.

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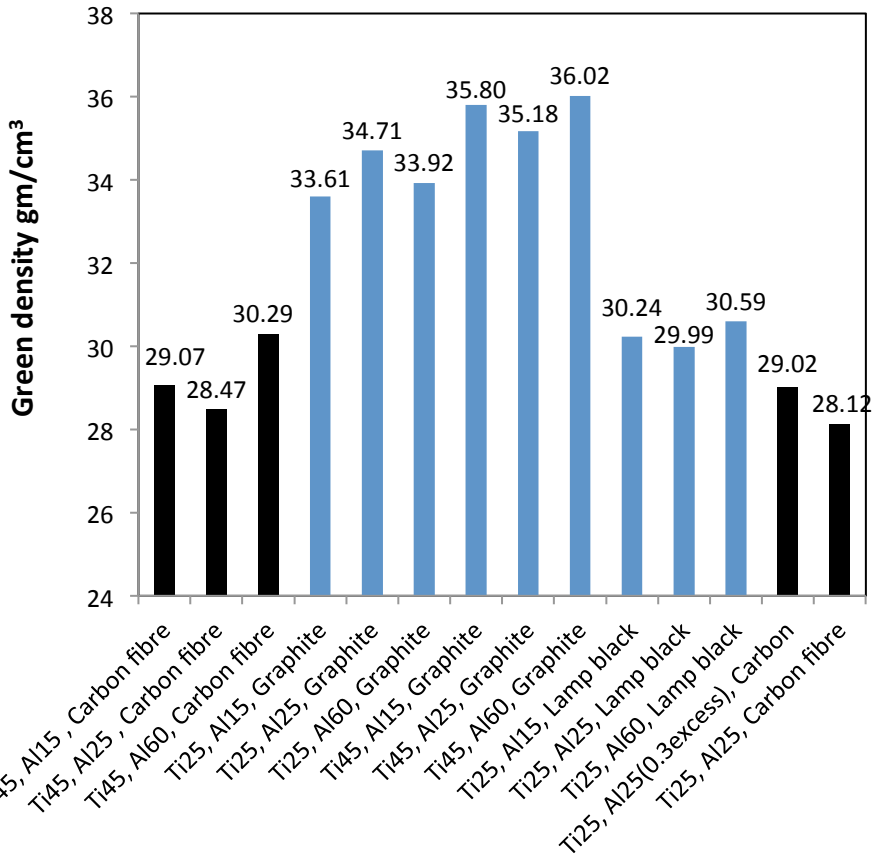

45µm, Al 25µm, Carbon fibre, (h) Ti 25µm, Al 25µm, Carbon fibre, (i) Ti 25µm, (0.3 mol excess) Al 25µm, Carbon fibre, and (j) Ti 25µm, Al 25µm, Lamp black

Figure 7 (a) Schematic of the combustion process showing the effect of graphite/lamp black on the final product formed (a) Schematic showing the combustion reaction image showing the formation of Ti_2AlC

Figure 8 XRD spectrum showing the evolution of compounds when excess Al is added to the reaction $2Ti + (1 + y)\ Al + C \rightarrow Ti_2AlC + yAl$. (a) y=0.1, (b) y=0.2, (c) y=0.3

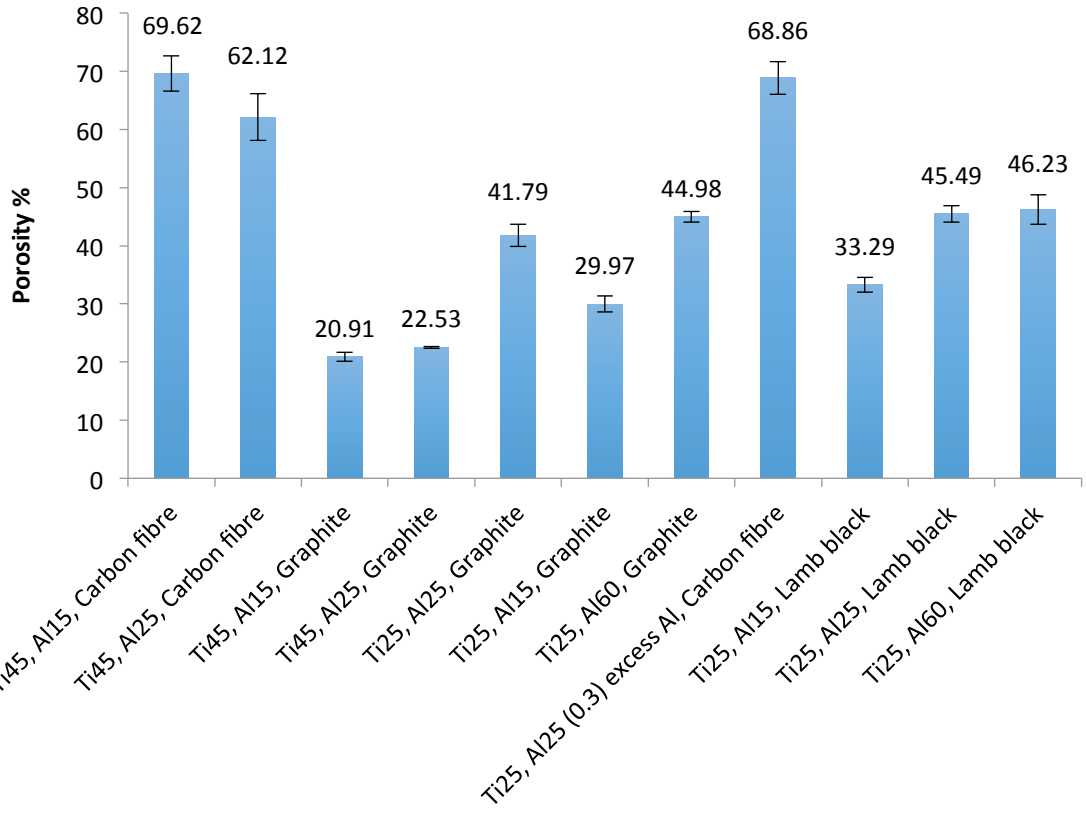

Figure 9 Porosity level (%) of the Ti_2AlC formed by SHS process from different combinations of particle sizes